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A CHARACTERIZATION OF V-391/H46-8B, A TOUGHENED BMI PREPREG SYSTEM PRODUCED BY U.S. POLYMERIC

CHARLES S. HILL
SRUCTURAL MATERIALS BRANCH
NONMETALLIC MATERIALS DIVISION

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WRIGHT RESEARCH AND DEVELOPMENT CENTER
AIR FORCE SYSTEMS COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OH 45433-6533

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CHARLES S. HILL, Proj Engr
Structural Materials Branch
Nonmetallic Materials Division

CHARLES E. BROWNING, Chief
Structural Materials Branch
Nonmetallic Materials Division

FOR THE COMMANDER

MERRILL L. MINGES
Director
Nonmetallic Materials Division

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The objective of this research effort is to characterize an advanced composite prepreg system. The material under investigation is U.S. Polymeric's V-391, a modified BMI resin, on continuous Hitex 46-8B graphite fibers. U.S. Polymeric has stated that the V-391 resin has the toughness of thermoplastics and the temperature performance of BMI's (Konarski 89). This study shows that although V-391/H46-8B shows improved toughness over baseline BMI's, its toughness is only a fraction of that of current thermoplastics, but its processability makes it an attractive material over baseline BMI's.

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FOREWORD

A training program has been developed at WRDC/MLBC to familiarize newly hired engineers with the fabrication and testing of fiber reinforced composites. The program results in the submission of a technical report for publication. This report is the result of the training program research which took place July 11 through September 14 at the Materials Laboratory of the Wright Research and Development Center located at Wright Patterson Air Force Base, Ohio. The work was performed by the author, Charles Hill, who was a third year co-op student from the University of Cincinnati, Department of Materials Science and Engineering, during his first cooperative education assignment.

ACKNOWLEDGEMENTS

This research effort would not have been possible without the contributions and guidance of the following members of the University of Dayton Research Institute, who carry out an on-site contract at the Materials Lab: Brian Rice, Bill Ragland, Ron Cornwell, Bill Price, Ken Lindsay, and Chuck Fowler. Their expertise and experience was invaluable to the timely completion of this effort.

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INTRODUCTION

The Structural Materials branch of the Wright Research and Development Center Materials Laboratory (WRDC/MLBC) has developed a training program in which new personnel fabricate and characterize a continuous fiber reinforced polymer matrix composite system. Usually the composite is being evaluated by the branch for potential use in Air Force systems. In order to gain knowledge about all aspects of composites, personnel conduct all procedures, from layup of the prepreg to evaluation of the data. This research was carried out as part of the MLBC training program by the author, a third year materials engineering co-op student, with the guidance of Brian Rice, an experienced engineer of the University of Dayton Research Institute (UDRI) on-site contractor for MLBC. The time period for this work was limited to a ten week period, the length of one co-op quarter, so that the training would not have to be continued upon returning from the school quarter. The test matrix was designed considering this time constraint and some compromises that were made will be discussed. The data that has been reported should be considered an initial material screening and is not intended for design purposes.

The composite being studied consists of a toughened or modified Bismalemide resin produced by U. S. Polymeric called V-391 and Hitex 46-8B graphite fibers, in a unitape prepreg system. The manufacturer claims the material is easily processed with epoxy like process cycles, has good hot-wet property retention up to 350° F, and has a substantial increase in toughness not previously available in high temperature matrices. This prepreg is being characterized as part of a comparative study of several toughened BMI systems which will be released in a later report.

BACKGROUND

The use of organic matrix composites in aircraft structural components is limited by their inability to retain superior mechanical properties at higher operating temperatures and in moist environments. Bismalemide resins were developed for use in higher temperature computer boards and later became popular as a resin capable of higher temperatures than epoxies. Since the late seventies, BMI resins have been used in aircraft and space structures. They are inherently brittle due to the highly crosslinked nature and high glass transition temperature of the polymer. A relatively low damage tolerance results from the brittle matrix. High toughness and damage tolerance has led to the popularity of thermoplastic matrices, which are difficult to lay up, having stiff prepregs, and are sensitive to solvents. The ease in processing of BMI systems with traditional methods and good prepreg drapeability, as well as the high temperature advantages, has driven research into the toughening of BMI resins to compete with the toughness of thermoplastics. Progress has been made by several producers in modifying, or toughening, the resins using new technology.

V-391 is a toughened BMI resin which the manufacturer, U.S. Polymeric, claims, "...combines the toughness of thermoplastics with the temperature performance of BMI's." and "...exhibits excellent composite toughness and mechanical properties while retaining the ability to be prepregged, handled, and cured with existing industry equipment designed for "350°F" epoxies." (Konarski, '89). A comparison of several modified BMI prepreg systems being conducted by Brian Rice led to the purchase of a variety of BMI prepregs. The sample batch of V-391/Hitex 46-8B prepreg which was investigated in this work had been shipped to Tobey Cordell at WRDC/MLBC. Techniques and equipment standard to MLBC material screenings were used in most of this research work.

EXPERIMENTAL PROCEDURES

A set of evaluative tests was used to characterize the material in both the cured laminates and uncured prepreg. These tests are commonly used at MLBC for similar characterizations, and throughout the description of the tests many references were made to previous technical reports.

Prepreg Analysis

Upon receipt from U.S. Polymeric, the prepreg was stored in a freezer at 0° F (-18° C) until testing could begin (three months). Visual and tactile evaluation revealed that the prepreg had good drape characteristics and slight tack. The prepreg was of good quality but some sections had wrinkles and separations. A yellowish substance on the surface was noted. It appeared to be a powder, and the possibility of it being a thermoplastic toughening agent was raised. However, the manufacturer's paper (Konarski 89) stated that it cures to a homogeneous single phase. Chemical evaluation of the substance was waived due to the proprietary nature of the material.

A Thermogravimetric analysis (TGA) was performed in the same manner on both the prepreg and cured laminate to determine the volatile content of each using a Dupont 951 Thermal Gravimetric Analyzer interfaced with an Omnitherm controller and software on an IBM PS/2 model 60. It was operated from room temperature to 600° C at a rate of 10° C per minute in a nitrogen atmosphere. Prepreg and laminate samples were also tested in a Dupont 910 Differential Scanning Calorimeter (DSC) with an Omnitherm Controller on the same computer. The DSC was run from 20° to 400° C at a rate of 10° C per minute in a nitrogen atmosphere to determine the heat of reaction and exotherm temperatures of both the prepreg and composite.

An RMS 7200 rheological characterization unit was used to determine the viscoelastic behavior of both the prepreg and the cured laminate. A ten ply unidirectional prepreg sample was cycled from room temperature to 400° C at a rate of 2° C per minute at a frequency of 100 radians per second. The storage and loss moduli as well as their ratio were plotted as a function of temperature. From this plot the glass transition temperature and "processing window" can be determined. A twelve ply cured sample was tested in the same manner to determine if it had fully cured.

Panel Fabrication

The prepreg roll was cut and laid up into the five panel configurations shown in Table 1. The bagging technique commonly used at MLBC was used, as shown in Figure 1. A Thermal Equipment Autoclave, model 8397, was used for the curing of the panels. Cure and post-cure cycles were provided by the manufacturer and are provided below.

V-391 cure cycle

Full vacuum and pressurize to 100 psi
Heat-up to 350° F at 5° F per minute
At 300° F vent vacuum bag
Hold at 350° F for 2 hours
Cool down at 5° F per minute

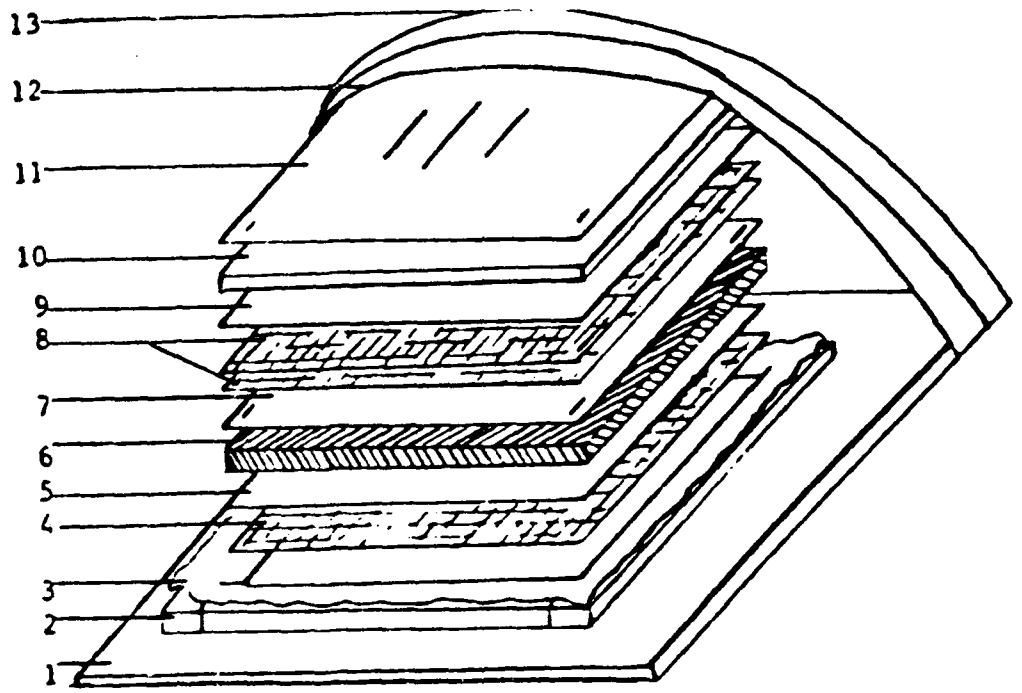
Post-cure cycle

Heat-up to 425° F at 5° F/min
Hold at 425° F for 6 hrs
Cool down at 5° F/min

TABLE 1

PANEL ORIENTATIONS

panel ID	# of plys	orientation	size [in]	comments
BR/39/167A	12	$[0^\circ]_T$	12"x12"	
BR/39/167B	8	$[+/-45^\circ]_{2S}$	12"x12"	
BR/39/167C	8	$[+/-45^\circ]_{2S}$	12"x12"	
BR/39/167D	24	$[0^\circ]_T$	12"x12"	release ply
BR/39/167E	12	$[+/-30^\circ,-/+30^\circ,90^\circ_2]_S$	9"x6"	
BR/39/167F	24	$[0^\circ]_T$	12"x6"	



1. Non-porous Teflon mold release on caul plate.
2. Silicon sealant tape dam.
3. Porous Teflon coated glass fabric; Sides are folded and taped to form a package, enclosing layers 4-11.
4. Bleeder cloth.
5. Porous Teflon coated glass fabric.
6. Laminate.
7. Porous Teflon coated glass fabric.
8. Bleeder cloth.
9. Nonporous Teflon coated glass fabric.
10. Caul plate.
11. Nonporous Teflon coated glass fabric.
12. Glass fabric.
13. Vacuum bag.

FIGURE 1

MLBC Bagging Technique (Wagner, 1989)

Physical Testing

All panels were evaluated by a series of tests to determine the quality of the cured laminate. Non-destructive C-scans were performed on a Testech Ultrasonic Immersion system, and no major voids or delaminations were detected in the panels. Representative sections of each panel were reserved for photomicrography, specific gravity, and fiber volume evaluations. An Olympus inverted stage light microscope and Polaroid film were used to take photomicrographs of mounted and polished samples from every specimen. The photographs are included in the results. Specific gravity was obtained analytically, using Archemedes principle, by weighing small samples in air and water. The resin was then decomposed in a heated flask of 99.9% Sulfuric acid over a period of two hours. The manufacturers data for fiber density was used in fiber volume calculations.

Mechanical Testing

Shown in Table 2 is the test matrix which was limited, due to lack of time and material, to a small number of standard mechanical tests. Detailed descriptions of the tests and sample dimensions can be found in (Carlin '87) or (Wagner '89) and the test methods will not be described in detail here. Standard tests used are shown in Table 2. The Mode I, Mode II, and edge delamination tests are not standardized to date. but are being reviewed by the ASTM D30 committee. The methods used are described in detail in (Carlin '87) and (Wagner '89). These tests give fracture toughness values and insight to the fracture behavior of the material, while the standard tests are used to determine elements in the compliance matrix.

TABLE 2 MECHANICAL TEST MATRIX

TEST	DRY				WET	
	RT	250°F	300°F	350°F	RT	300°F
0° 4-Pt. Flex	5	5	5	5	5	5
0° 3-Pt. Flex	5					
90° 4-Pt. Flex	5					
0° 4-Pt. Shear	5	5	5	5	5	5
0° Tension	5					
+/- 45° In plane shear tension	3			3		
Edge Delamination	5					
Mode I	5					
Mode II	5					

A diamond impregnated saw was used to cut out the various test specimens required by the test matrix. Specific dimensional specifications are listed in Table 3. All specimens were true to 0.001 in/in parallel. Mode I and Mode II test coupons were produced with a 1.5" wide Teflon release ply, placed inside the top edge of the zero degree coupons, half way through the thickness, as a crack initiator. Edge delamination specimens were polished on one edge with decreasing grits so that stress at initial delamination could be detected. All tension type tests, including edge delamination, were tabbed with 1.25in, polyester, beveled tabs. All specimens were dried in a vacuum oven and stored in a desiccator.

TABLE 3. STANDARD TEST DIMENSIONS

Test	ASTM standard	length	width	thickness
0° 4-Pt. Flex	D790-81	3.0"	0.5"	0.07"
0° 3-Pt. Flex	D790-81	3.0"	0.5"	0.07"
90° 4-Pt. Flex	D790-81	3.0"	0.5"	0.07"
0° 4-Pt. Shear	D790-81	3.0"	0.5"	0.14"
0° Tension	D 3039-76	9.0"	1.0"	0.07"
+/- 45° In plane shear tension	D3518-76	12.0"	1.0"	0.04"
Edge Delamination	D30	9.0"	1.0"	0.07"
Mode I	D30	9.0"	1.0"	0.14"
Mode II	D30	9.0"	1.0"	0.14"

Data on the moisture absorption and wet properties of the composite were obtained using two different methods. A water boil was used, along with the standard soak method commonly used at MLBC, henceforth refered to as normal moisture ageing (NMA), so that samples could be tested wet during the short co-op period. Wet data from twenty samples was obtained by a water (distilled) boil in a flask equipped with an electric heater and condenser. A 48 hour boil was planned, but the twenty-four ply shear specimens (F1-F10) showed significantly less moisture absorption than the twelve ply flex specimens after 48 hours, so a modified boil schedule was followed including 72 and 96 hour boils. Table 4 shows the boil data. The wet masses are given at each time they were weighed, and the final percent gained shown is the percent of mass gained in moisture when tested. The letters in the specimen ID notation (F1-A40) indicate the panel the samples were taken from and the numbers were each individual coupon. Although moisture aging provides good insight to the maximum moisture absorption by the material, the boil method was chosen because the long period of time required for aging and frequent weighing was not feasible for this co-op period.

Later, normal moisture aging and moisture aged wet testing was carried out by UDRI staff. The results of the moisture aging show that the water boil was not sufficient for sample saturation. The average absorbtion by the water boil method was 0.821% while the average for the standard soak method was 1.32%, after soaking at 160 °F for approximately fifty-eight days. The rate of weight gain had slowed but not stopped, so it is concieveable that the samples could have absorbed more water, but saturation beyond this point probably would not occur in normal use in aircraft applications. The difference in moisture absorption also relates to a difference in wet mechanical properties as can be seen in the mechanical test results. A plot of moisture absorption versus time has been included in Appendix A (Figure 38).

TABLE 4. WATER BOIL DATA

specimen	mass dry	mass wet 48h	mass wet 96h	total mass gained	percent gained
F1	6.1537	6.1866	6.2006	0.0469	0.76214
F7	6.4086	6.4433	6.4587	0.0501	0.78176
F10	6.4723	6.509	6.524	0.0517	0.79878
A31	2.755	2.7778		0.0228	0.82758
A32	2.6197	2.6413		0.0216	0.82452
A33	2.8013	2.8233	mass wet 72h	0.022	0.78534
A37	2.7866	2.8084	2.8115	0.0249	0.89356
A40	2.7893	2.8106	2.8143	0.025	0.89628
				average % gained=	0.82124

The 3-point and 4-point flex and 4-point shear tests were run on an Instron 1123 table-top load frame using the 4-pt. flex fixture described by Carlin (1988) and the crosshead in compression at a displacement rate of 0.05 in/min. The surfaces of the 90° 4-Pt. flex specimens were strain gaged on the tensile face. Strength and moduli were calculated using linear elastic beam theory which can be referenced in most strength of materials texts. The equations used are listed in Table 5.

TABLE 5. FLEX AND SHEAR MECHANICAL PROPERTY EQUATIONS

Property	Strain Measurement	3-Point	4-Point
σ_{max}	crosshead disp.	$3PL/(2bt^2)$	$3PL/(4bt^2)$
τ_{max}	crosshead disp.	$3P/(4bt)$	$3P/(4bt)$
E_{flex}	crosshead disp.	$(dP/du)L^3/(4bt^3)$	$(dP/du)L^3/(8bt^3)$
E_{flex}	strain gauge	N/A	$(dP/d\varepsilon)3L/4(bt^2)$

P=maximum load
L=support span
b=specimen width
t=specimen thickness
dP/du= slope of load vs. crosshead displacement curve
dP/dε=slope of stress-strain curve

The 0° Tension, In-Plane Shear (IPS), and Edge Delamination (ED) strengths were measured on an Instron TTS universal testing machine in tension. The strain measurements were taken using a high-strain magnification extensometer for tension and ED tests, but surface strain gages were used for the longitudinal and transverse strain measurements on the IPS tests. The following equations were used for calculating strength and moduli:

0° Tension	$\sigma_{max}=P/bt$	$E=dP/d\varepsilon(bt)$
G_{12}=IPS modulus	$G_{12}=P/2(\varepsilon_1-\varepsilon_2)bt$	$G_{12}=\tau_{max}/\nu$, $\tau_{max}=\sigma_{max}/2$, $\nu=\varepsilon_1-\varepsilon_2$

ν=poisson's ratio and ε_1 and ε_2 are the longitudinal and transverse strains respectively.

The interlaminar fracture toughness of the composite was tested in two modes: Mode I using the double cantilever beam (DCB) geometry, and Mode II using the end notched flexure (ENF) geometry. The methods used at MLBC for the DCB and ENF tests have been described elsewhere (Whitney et al., 1982) and the equations are shown in (Curliss, 1988). Identical methods were followed, as with all other mechanical tests, therefore, a detailed description of these methods is not necessary in this report.

RESULTS AND DISCUSSION

Physical Properties Data

Visual and tactile evaluation of the prepreg revealed good processing quality including: superior drape, slight tack, and few winding defects. The yellow substance visible on the surface, believed to be a toughening agent was examined with a JEOL LSM 840 Scanning Electron Microscope (SEM), which revealed that the substance was not a powder as it appeared but a resin-like substance. SEM micrographs of the prepreg at various magnifications are shown in Figure 3.

Thermal analysis of the prepreg produced the TGA scan shown in Figure 4. The scan indicates that two separate weight loss mechanisms are present, the first beginning at 185 C and ending at 251° C with a 1.54% weight loss. This mechanism is most likely a solvent added to increase tack and should volatize below the 177° C maximum cure temperature in the vacuum of the autoclave, thereby preventing void formation. The second, a 22% weight loss, begins at 382° C and ends at 438° C which is not of interest because it is far above the processing temperatures. TGA of the postcured laminate (Figure 5) gave a 20% weight reduction between 374° C and 424° C indicating that all solvents were extracted during processing.

A DSC scan of the prepreg is shown in Figure 6; integration of the peak gives a heat of reaction of 35.46 mcal/mg as shown on the plot. Figure 7 shows the DSC scan of the cured laminate. Lack of an appreciable exotherm shows that no further reactions occur after processing and that no moisture was present in the laminate. Thermomechanical Analysis (TMA) of the post cured sample was run using a Dupont TMA 800 with an Omnitherm data acquisition system (Figure 8). The TMA indicates a glass transition temperature of 220° C. The rheological behavior of the prepreg and laminate are illustrated in the Rheometric Dynamic Spectrometer (RDS) scans of Figure 9. The storage modulus, loss modulus, and tan delta of the torsion bar specimens are plotted. The decrease in the storage modulus of the cured laminate around 200° C corresponds to the softening that occurs as the resin approaches its glass transition temperature.

Ultrasonic C-scans of all the panels showed that they were free of all major voids. Voids were visible, however in the photomicrographs of panel C. This is most likely due to the fact that the photomicrograph sample was taken from an area near the edge of the panel. Voids would be more apparent there due to the flow of volatiles out the edges of the panels from between the plies. These voids could be due to such a short cure cycle but was not a problem. Photomicrographs of each cured panel are shown at various magnifications in Figures 10-26. Resin rich areas are apparent between plies and could possibly be due to the yellowish powder substance. lighter areas can be seen between plies figures 13,18,19, and 23. In these figures it seems that the substance is preventing the plies from coming together. The use of variable layers of bleeder plies is used to control resin flow and fiber volume. A test run using many bleeder plies could help to determine if the substance is indeed preventing complete consolidation, but this was not performed due to the lack of available material.

Results of specific gravity and fiber volume tests are shown in Table 6. Acid digestions were run at various times to ensure that fibers were not degraded by the sulfuric acid and it was determined that two hour acid digestion runs would give the most accurate fiber volume data.

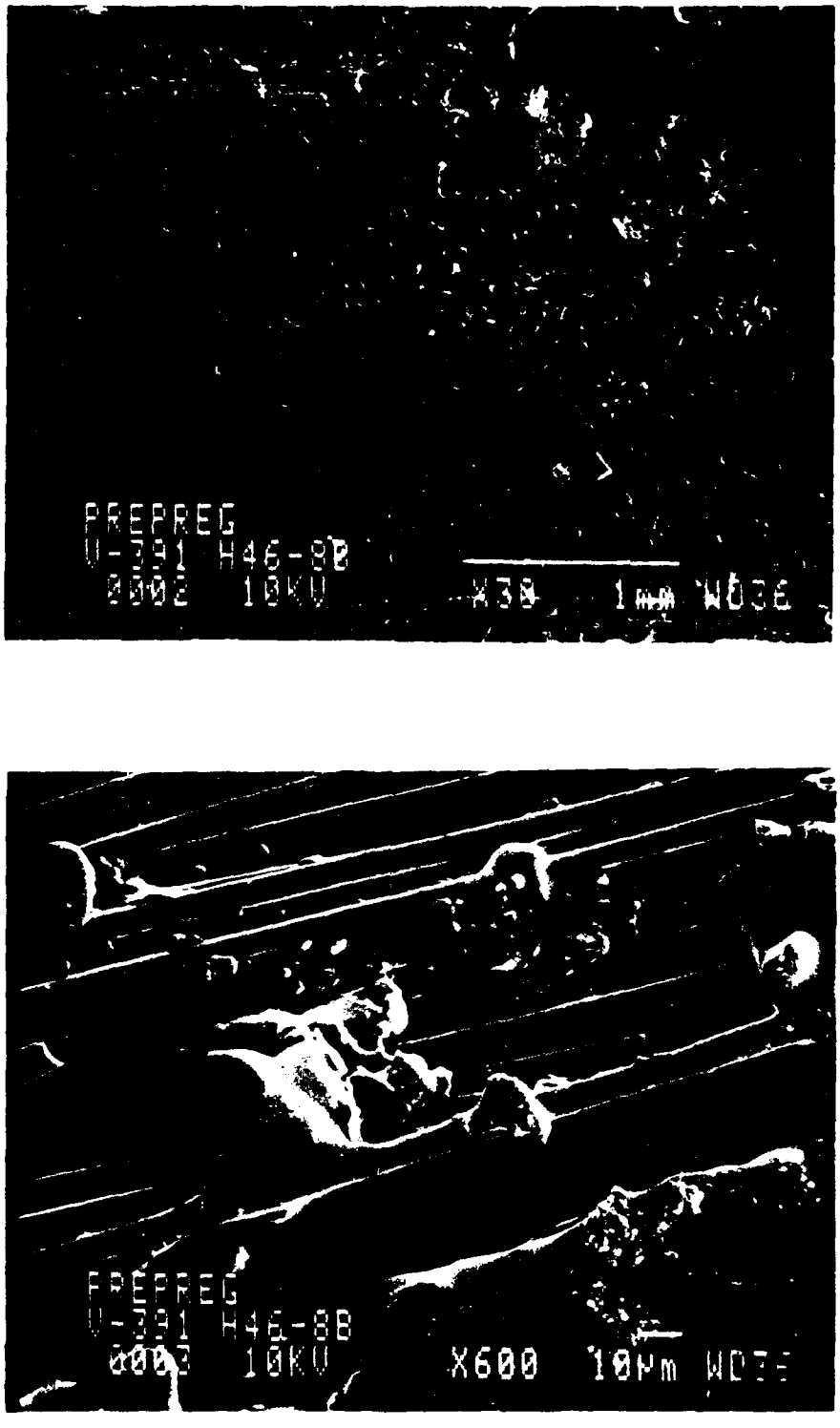


Figure 3. SEM Micrographs of V-391 prepreg at various magnifications

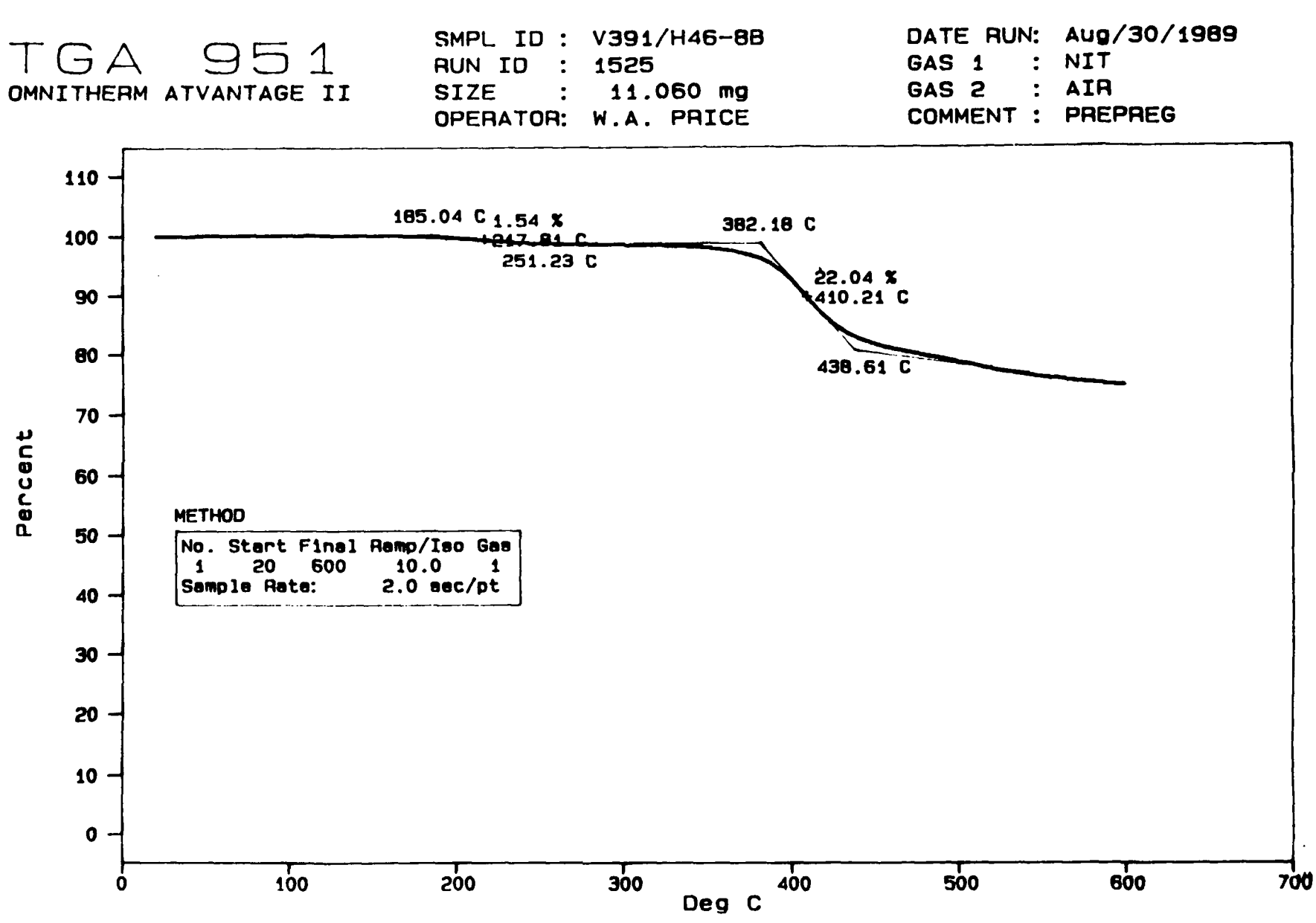


Figure 4. TGA plot of V-391 prepreg

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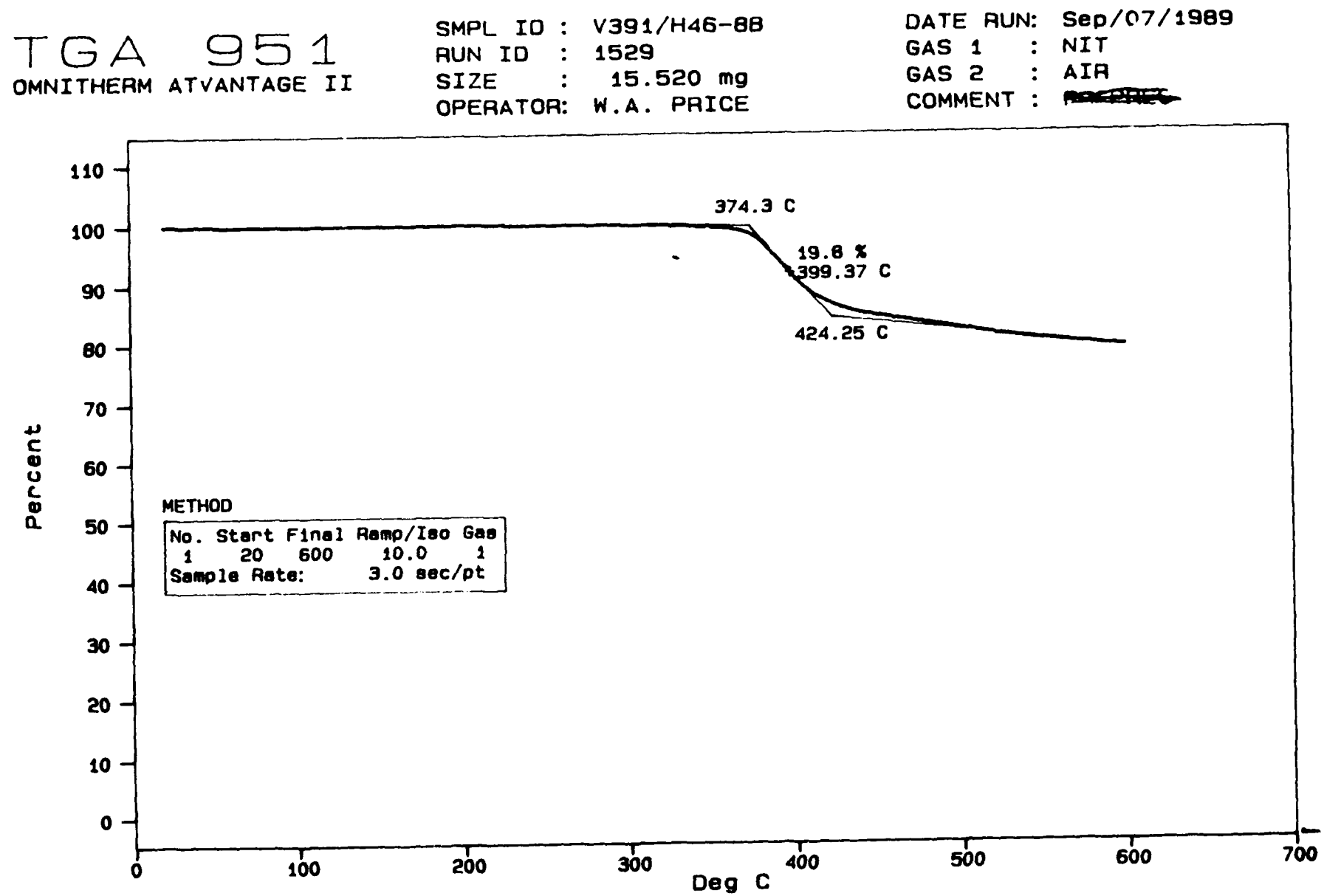

Figure 5. TGA plot of postcured composite

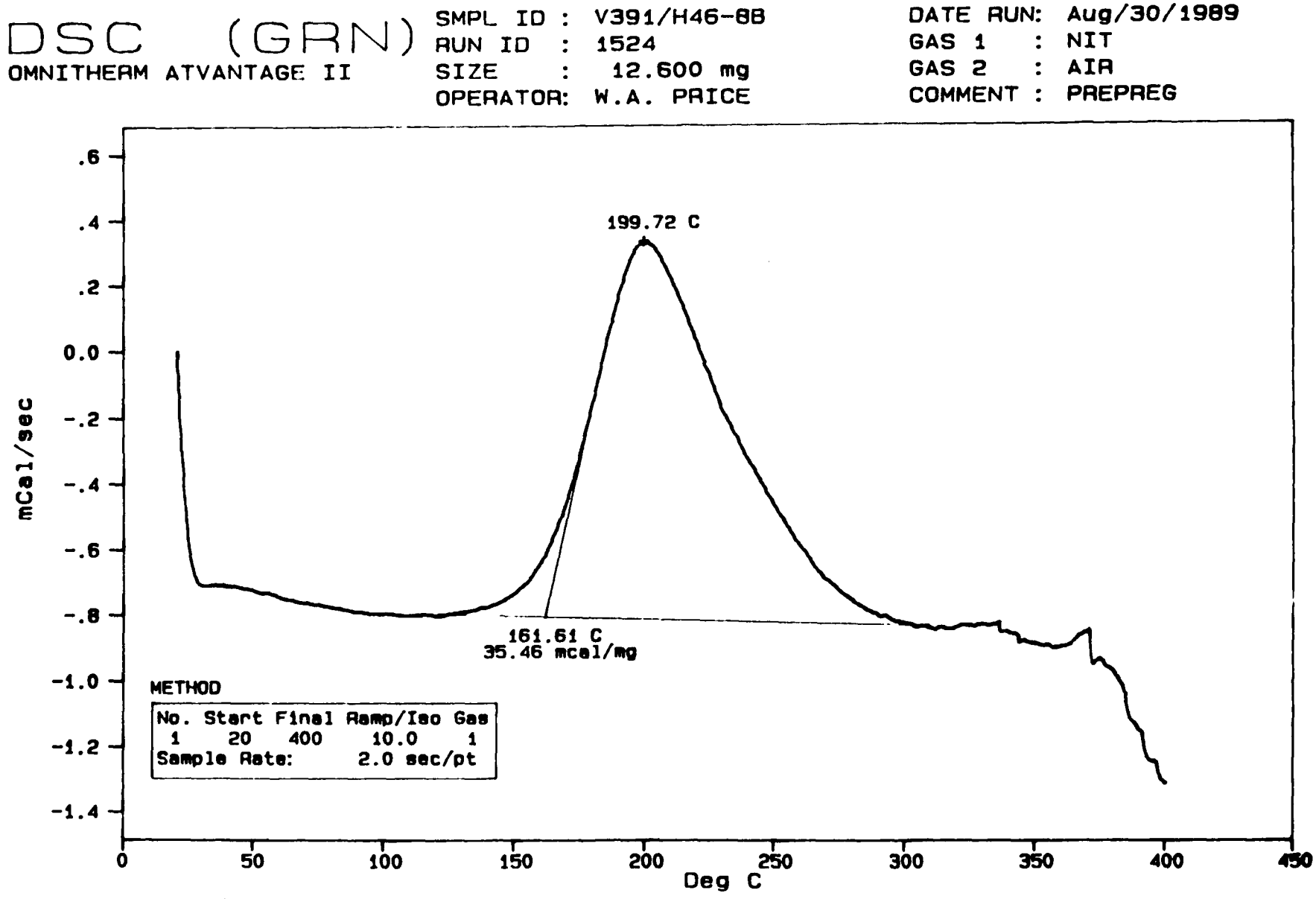

Figure 6. DSC plot of V-391 prepreg

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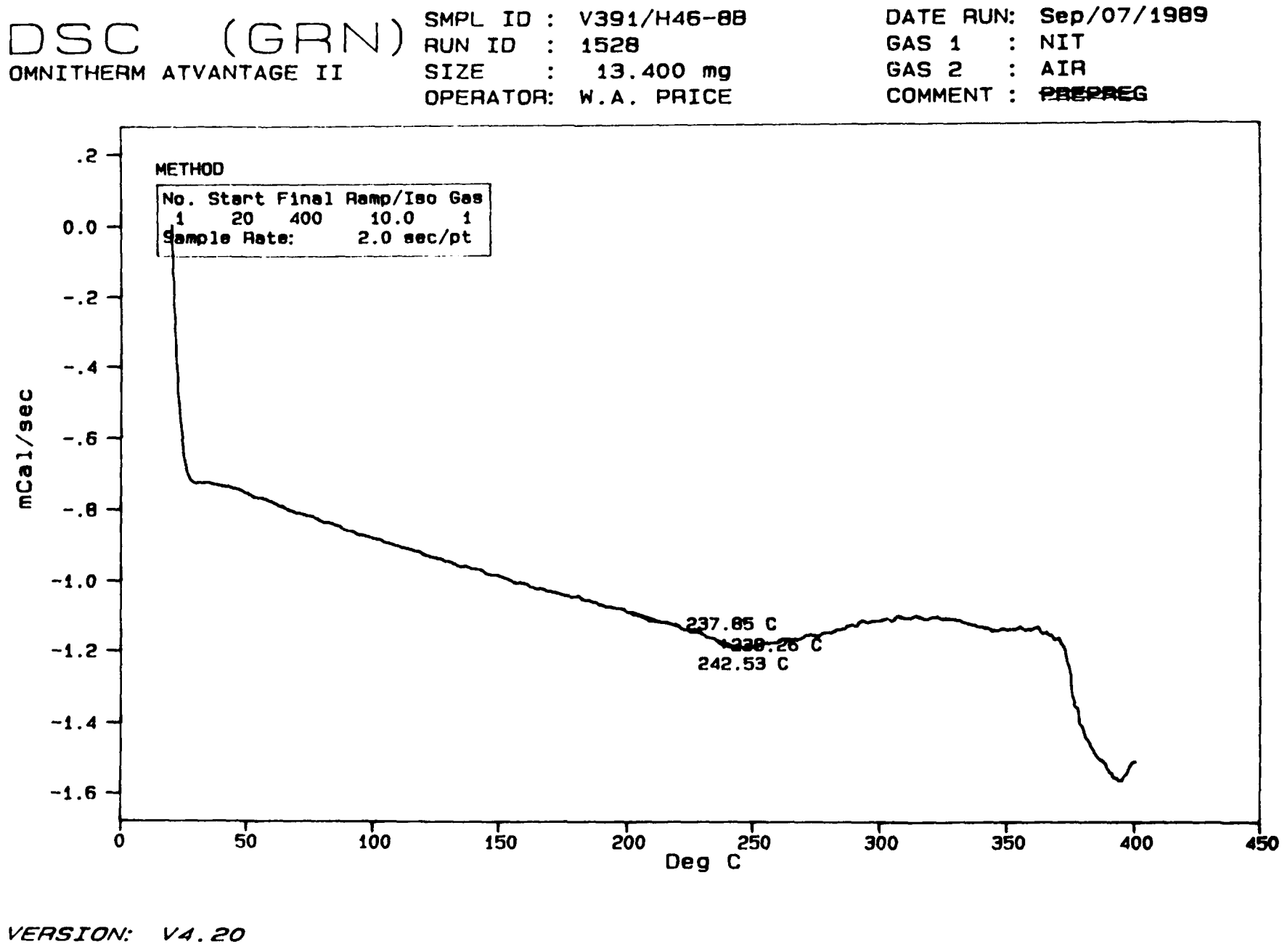


Figure 7. DSC plot of postcured composite

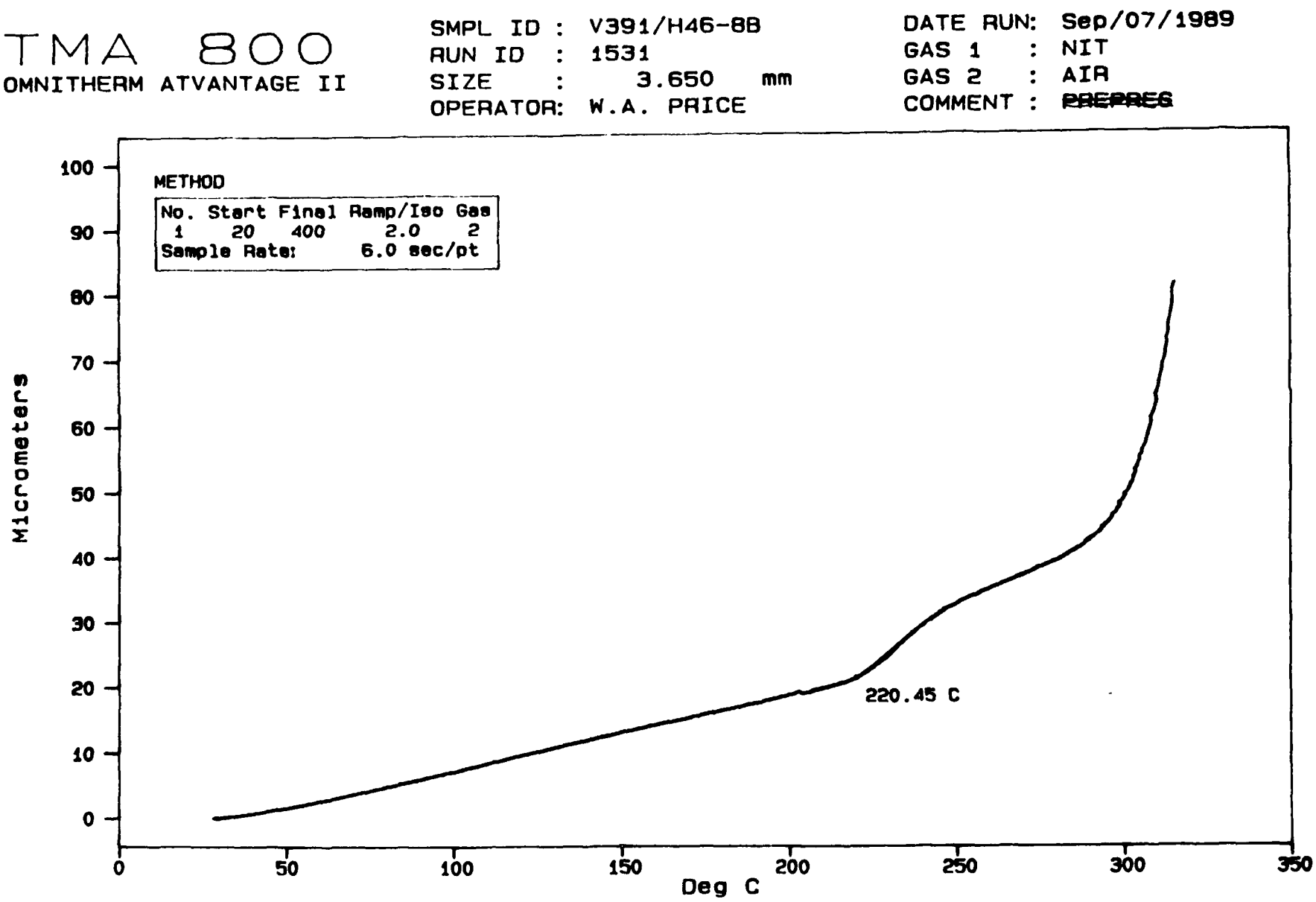


Figure 8. TMA plot of postcured composite

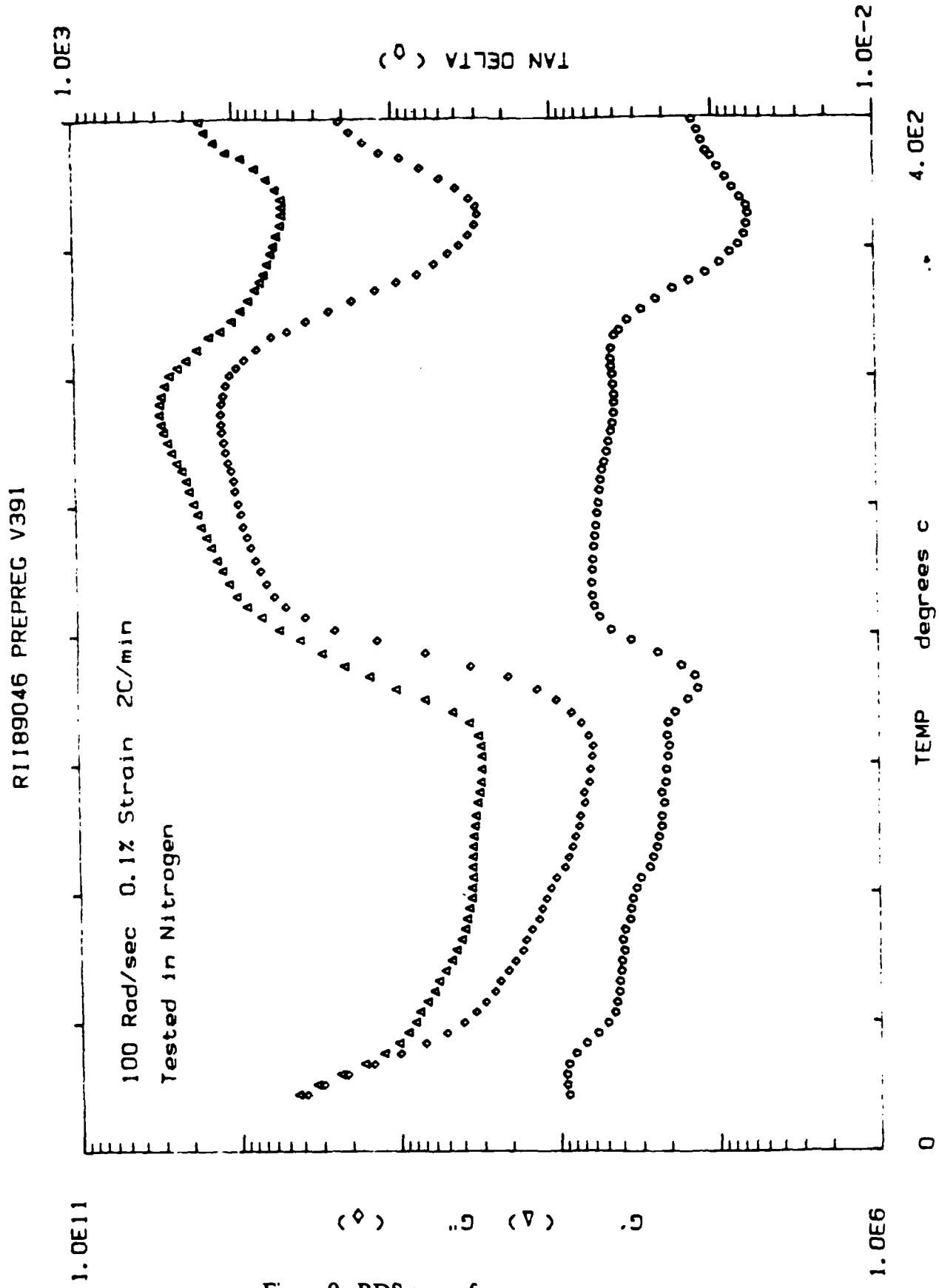


Figure 9. RDS scan of prepreg

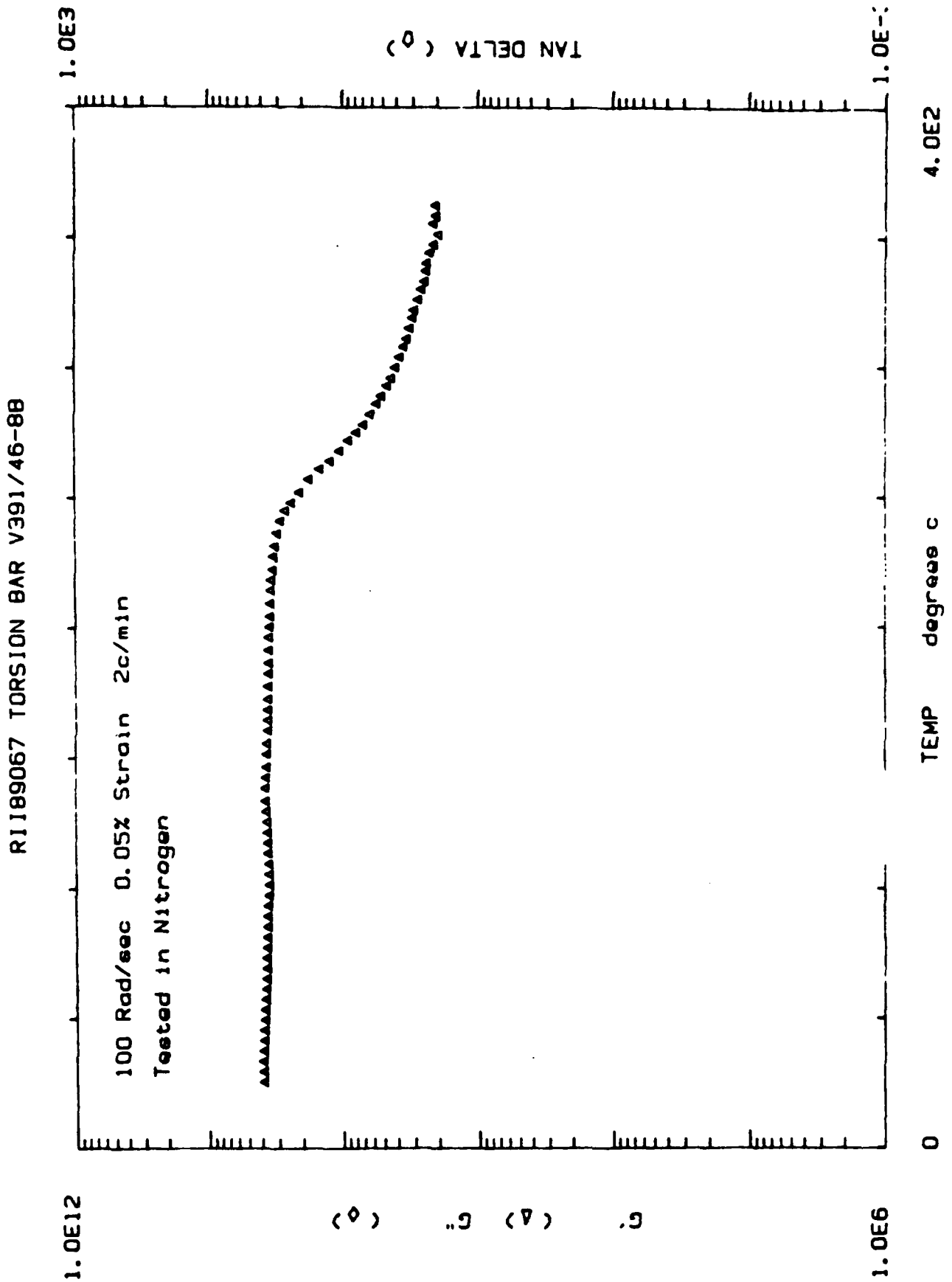


Figure 10. RDS scan of V-391 postcured composite

Photomicrographs of Panel A $[+/-45^{o}]_{S}$, 8ply

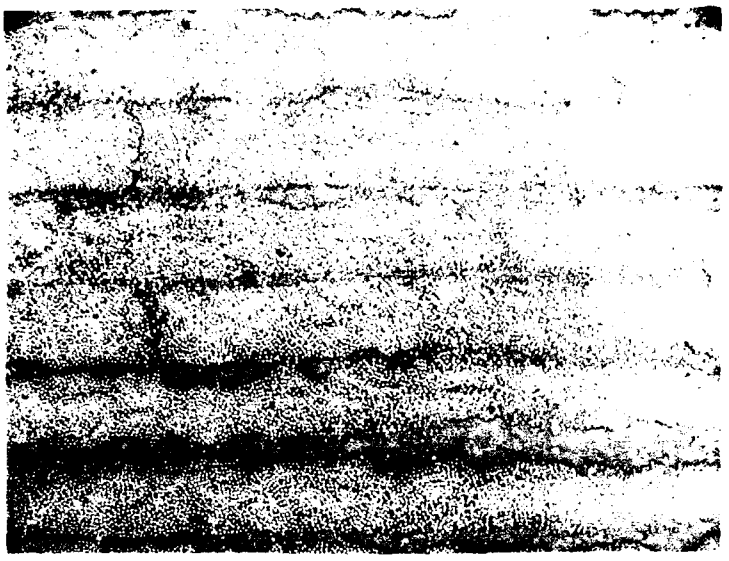


Figure 11. 75X

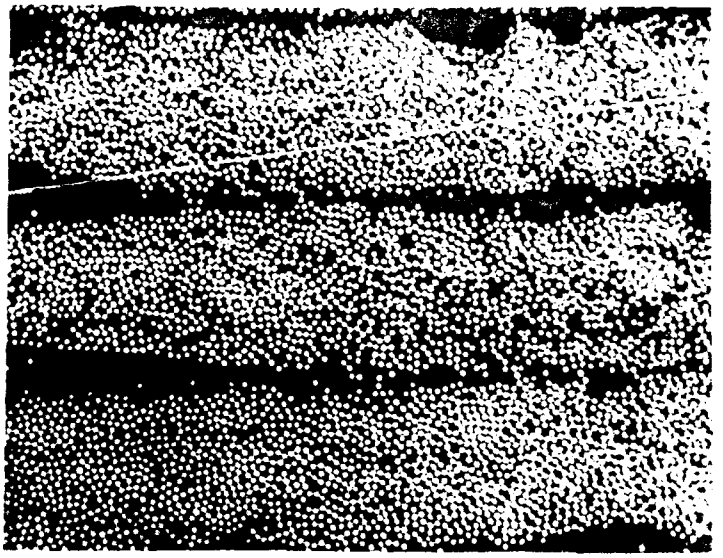
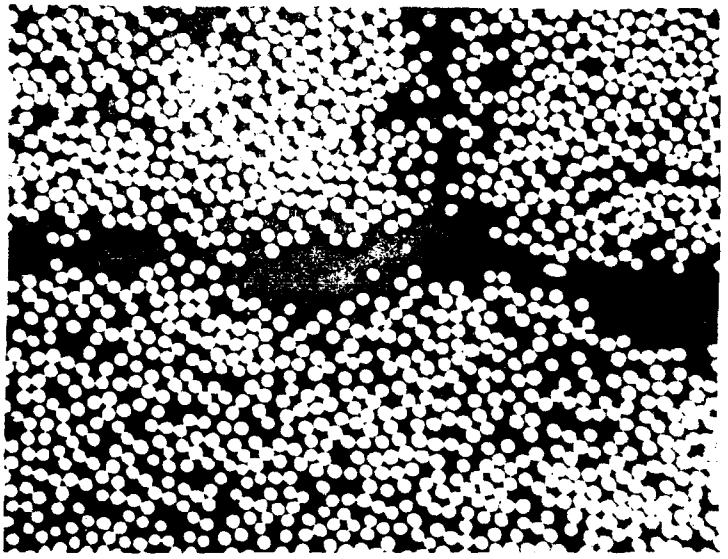


Figure 12. 150X

Figure 13 300X



Photomicrographs of Panel C [0°], 8 ply

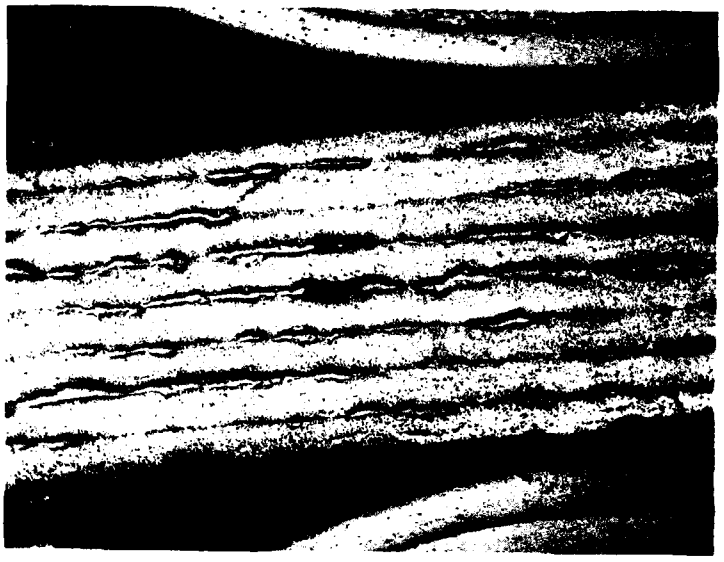


Figure 14. 37.5X

Figure 15. 75X

Figure 16. 300X

Photomicrographs of Panel D [0°], 24 ply

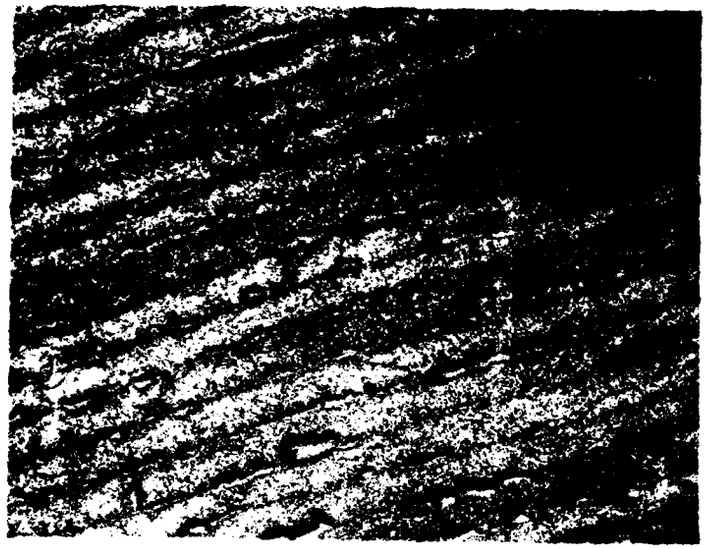


Figure 17. 37.5X

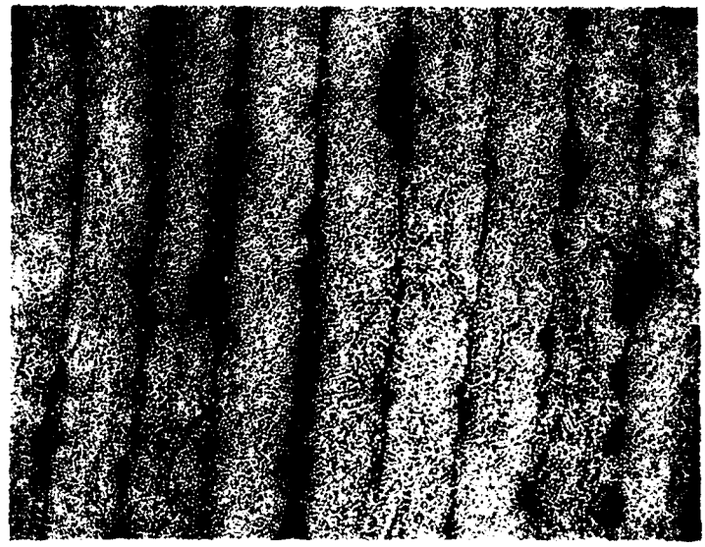
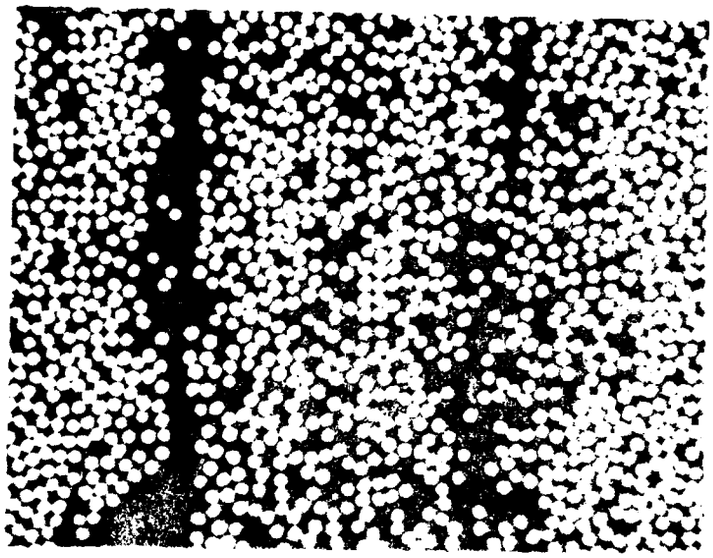


Figure 18. 75X

Figure 19. 300X



Photomicrographs of Panel E [+/-30°,-/+30°,90°2]s,12 ply

Figure 20. 37.5X

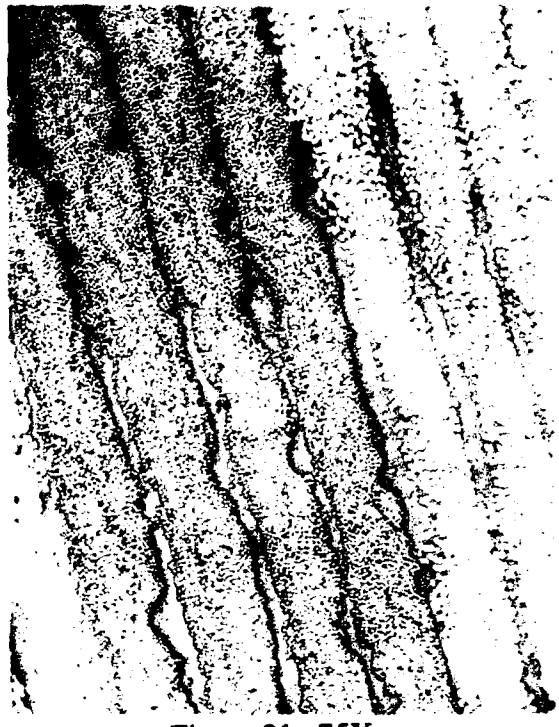


Figure 21. 75X

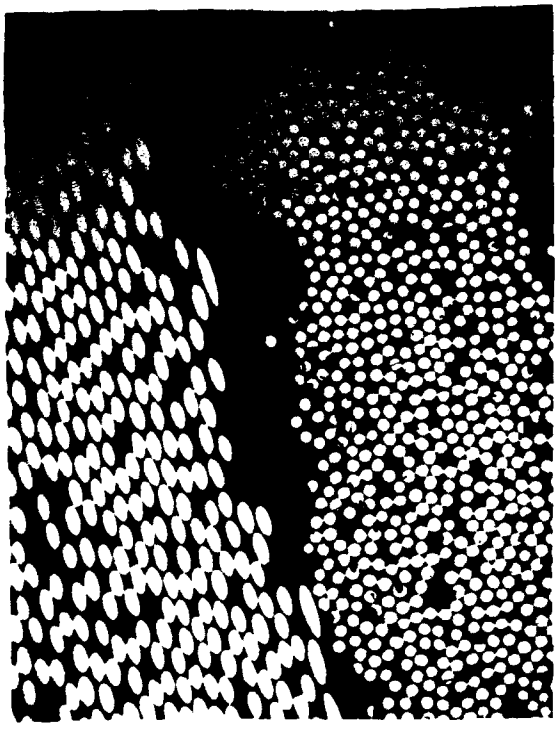


Figure 22. 150X

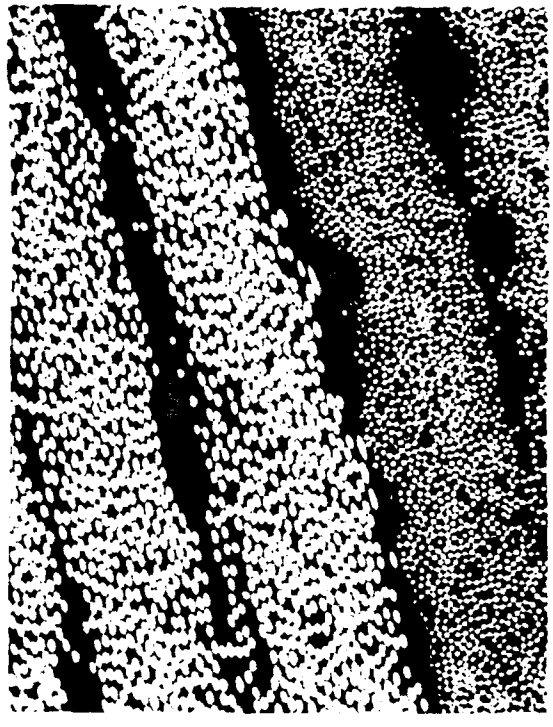


Figure 23. 300X

Photomicrographs of Panel F [0°],24 ply

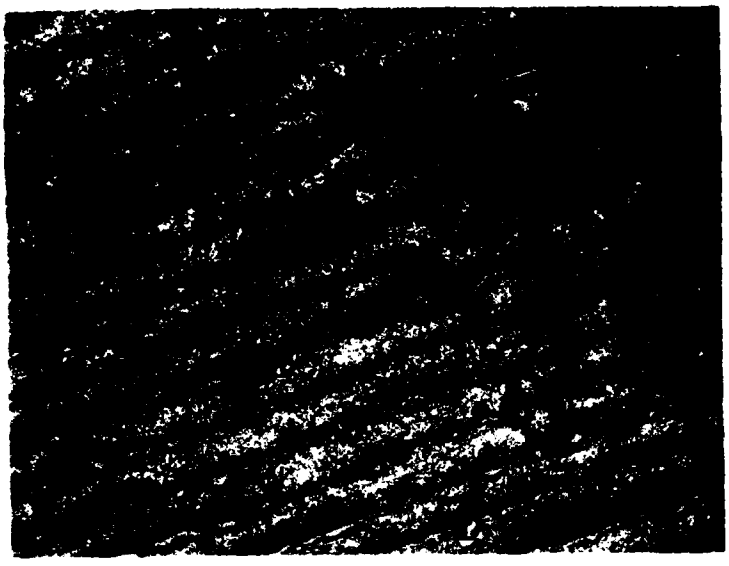


Figure 24. 37.5X

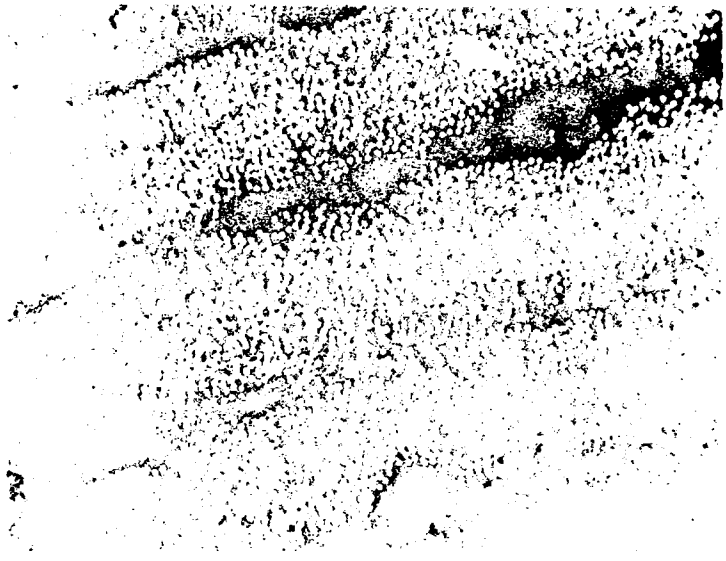


Figure 25. 150X

Figure 26. 300X

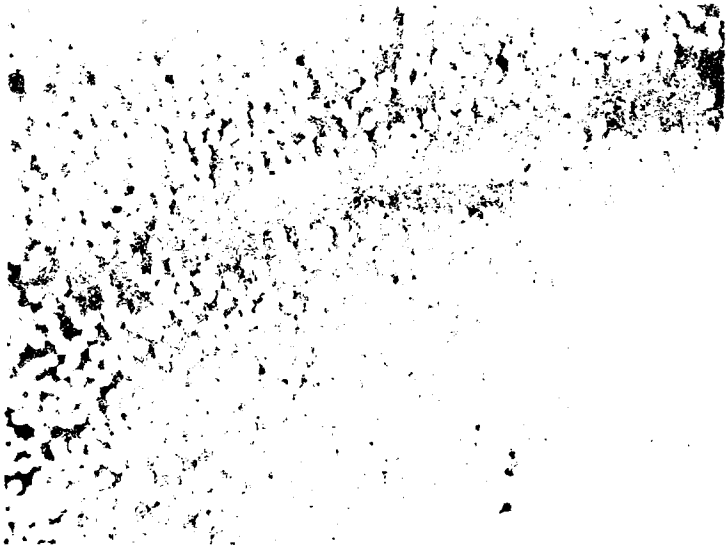


Table 6. Specific gravity and fiber volume data for each panel

Panel ID	Specific Gravity of Specimen	Density of Fiber (from manufacturer)	Percent Fiber of specimen (by volume)	Length of Acid Digestion (hrs)
A	1.6	1.81	54	2
B	1.5	1.31	51	2
B	1.5	1.81	48	3
B	1.5	1.81	45	4
C	1.5	1.81	53	2
D	1.5	1.81	54	2
E	1.6	1.81	56	2
F	1.5	1.81	56	2

Mechanical Properties Data

Table 7 summarizes the mechanical test results. The percent fiber volume and percent moisture gain are included in this table for easy access. The failure modes are also included. The averages (usually of five specimens)[1] of each test (row) are shown for each applicable property (ie. strength, modulus, etc.). The percent retention in strength from the dry room temperature property is provided to show the drop-off of strength as temperature increases and moisture is introduced. Appendix A includes plots of 4-point flex data so that the affects of hot/wet conditions can be visualized. Mechanical test results were typical of modified BMI composites. An interesting feature however, is that an increase in strength was noted between the RT dry and RT wet tests. The results show that the flex strength at RT, dry is 1368 MPa and that of RT, wet is 1610 MPa.

Examination of fracture surfaces was completed on the JEOL LSM 840 SEM. Figures 27-31 illustrate resin adhesion to the fiber in some failure modes. Figures 27 and 28 show pulled out fibers from a flex test, and figures 29 and 30 show fractured fiber ends at the resin-fiber interface. Hackles are evident on 4-pt shear failure surfaces (Figures 32 and 34) and edge delamination surfaces (Figure 33), due to the resin rich areas between plies peeling up during interlaminar shear failures . Total fracturing of fibers was seen in compressive failures, due to stress concentration at the support pins during some 4-pt flex and shear tests: Figure 35 shows a 4-pt flex specimen that failed in compression under the support pin, Figures 36 and 37 show 4-pt shear specimens that failed in compression. The magnifications and sample numbers are shown on the photographs.

A 4-pt flex strength of 198 KSI is 80% of the 3-pt flex strength of 246 KSI, this difference is most likely due to a difference in failure mode. The 3-pt and 4-pt flex tests are used for various reasons and show different behavior for diffenent materials. The lower 4-pt flex may be due to more stress concentration at ther lower support pins, due to the load being spread out, and the compressive failure occuring on the tension side of the specimen lowers its strength, while in the 3-pt flex compressive failure occurs at the upper indenting pin on the surface in compression, leaving the fibers near the tension surface intact. The RT 90° 4-pt flex strength of 12 KSI was only 6% of the RT, dry, 0° 4-pt flex strength of 198 and this gives some indication of the resin properties. A 50% drop from a RT, 90° 4-pt flex strength of 12 KSI to a 350° F 90° 4-pt flex strength of 6 KSI indicates a 50% drop in resin properties at 350° F. Good retention in strength is noted in the dry 0° 4-pt flex tests up to 350° F, but the wet tests show large drops in strength at 300° F: 66% retention in the wet 300° F 4-pt flex and 60% retention in the NMA 300° F 4-pt flex test.

[1] The number of data points for each value varies and is shown in the test matrix (table 2).

TABLE 7. Completed Test Matrix for V-391/H46-8B Composite

TEST	% fiber by volume	moisture gain (%)	DRY WET	Temp [F]	Temp [C]	strength [KSI]	strength [MPa]	Modulus [MSI]	Modulus [GPa]	% retention in strength from RT,dry	Failure Mode
0 4PF	54		dry	74	23	198	1368	18.3	126	100	[1]
	54		dry	300	149	179	1236	18.4	127	90	[1]
	54		dry	350	177	171	1177	20.9	144	86	[1]
	54	0.81	wet	74	23	234	1610	20	138	117	[2]
	54	0.89	wet	300	149	131	906	18.6	128	66	[2]
0 4PF	54	1.32	NMA	RT	RT	202	1393	20.9	144	102	[2]
	54	1.32	NMA	350	177	120	827	18.1	125	60	[2]
0 3PF	54		dry	74	23	246	1698	20.9	144		[2]
90 4PF	54		dry	74	23	12	80	1.2	8	100	[5]
	54	1.32	NMA	RT	RT	6	41	1.25	9	52	
0 TENSION	54		dry	74	23	266	1831	20.7	143		
+/-45 TENS	53		dry	74	23	17	114	0.74	5	100	[6]
	53		dry	350	177	13	88	0.73	5	77	[6]
						stress at delamination		ultimate stress			
						[Ksi]	[MPa]	[Ksi]	[MPa]		
EDGE DELAM.	56		dry	74	23	32.20	222	57.20	394		[9]

TEST	% fiber by volume	moisture gain (%)	DRY WET	Temp [F]	Temp [C]	ILS Strength [Msi]	ILS Strength [GPa]		% retention in strength from RT,dry	failure mode
0 4PS	54.3		dry	74	23	12.6	86.874		100	[3]
	54.3		dry	250	121	10.2	70.327		80	[3]
	54.3		dry	300	149	9.1	62.742		72	[3]
	54.3		dry	350	177	7.96	54.882		63	[3]
	56.13	0.78	wet	74	23	10.5	72.395		83	[3]
	56.13	0.78	wet	300	149	6.64	45.781		53	[3]

TEST	% fiber by volume	moisture gain (%)	DRY WET	Temp [F]	Temp [C]	Interlaminar fracture toughness [lb*in/in^2]	Interlaminar fracture toughness [KJ/m^2]		failure mode
MODE I	54		dry	74	23	1.57	0.27		[7]
MODE II	54		dry	74	23	4.14	0.73		[8]

[1] Mixed mode: compression and interlaminar shear
[2] Compression
[3] Interlaminar shear
[4] Compression buckling
[5] Tension
[6] In plane shear
[7] Mode II delamination
[8] Mode I delamination
[9] Mixed mode: Modes I and II delamination

Figures 27 - 40. SEM micrographs of fracture surfaces

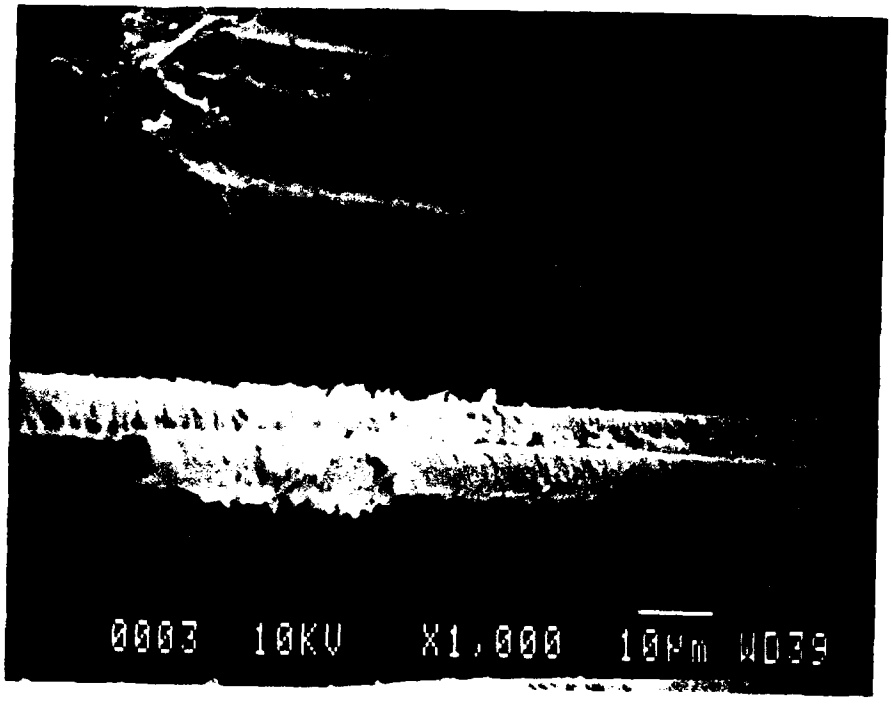


Figure 27

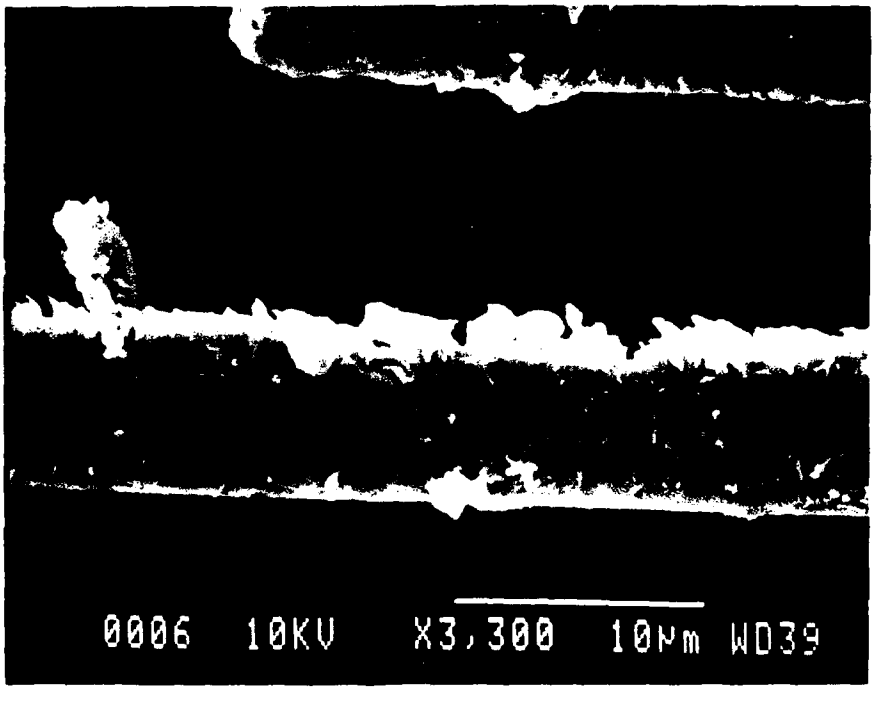


Figure 28

Figure 29

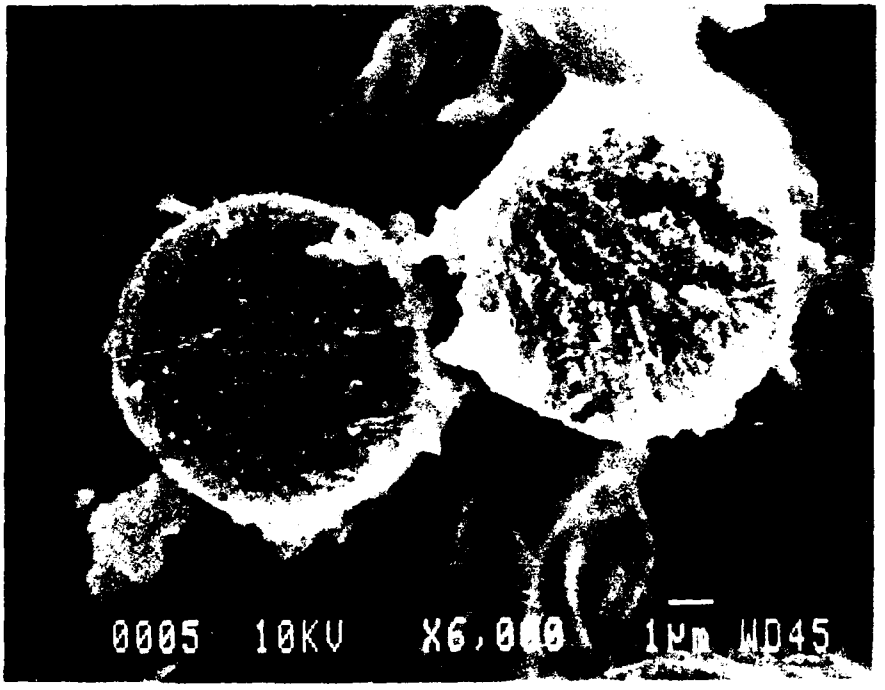


Figure 30

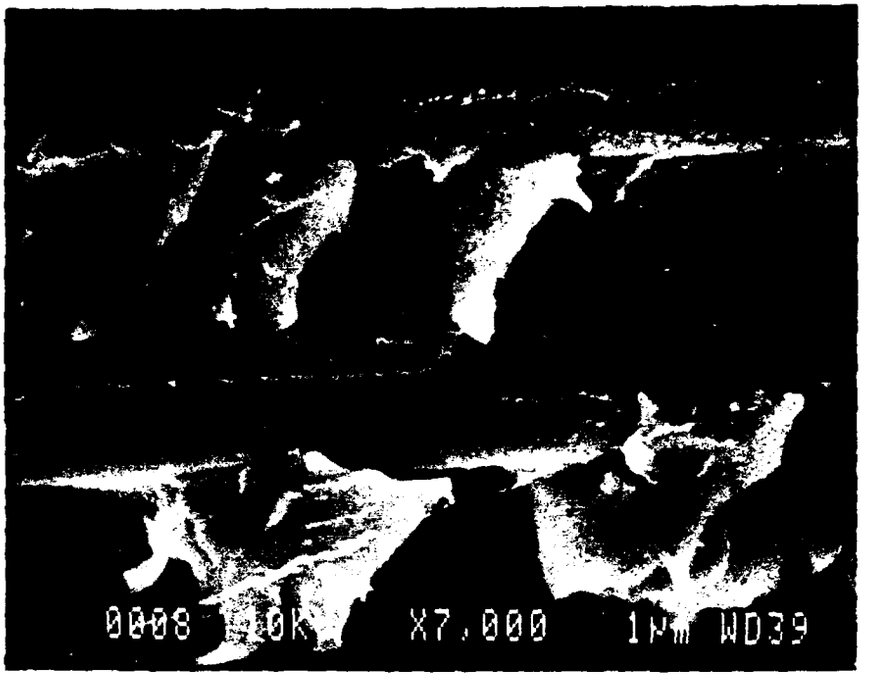


Figure 31

Figure 32

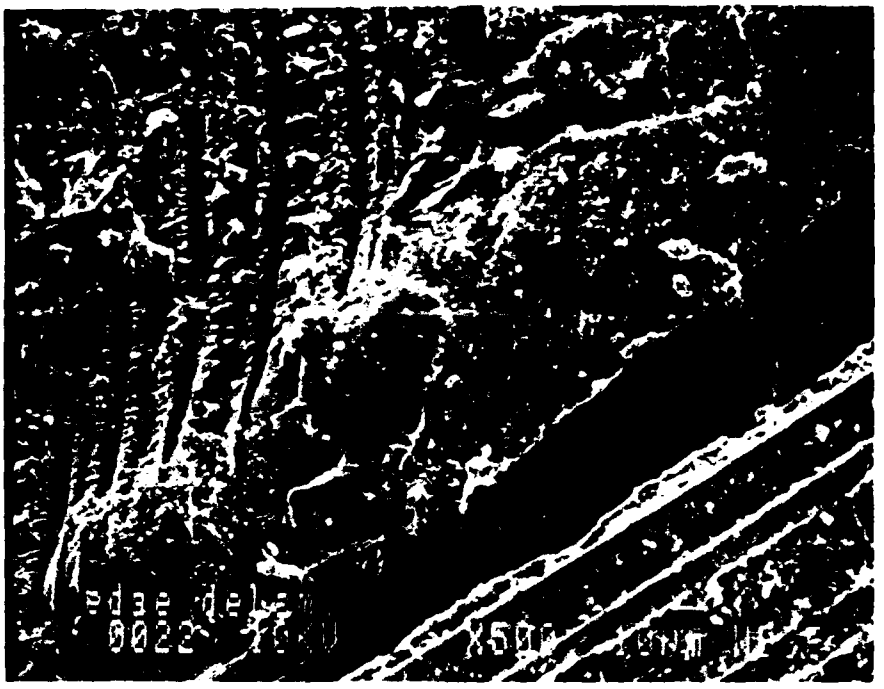


Figure 33

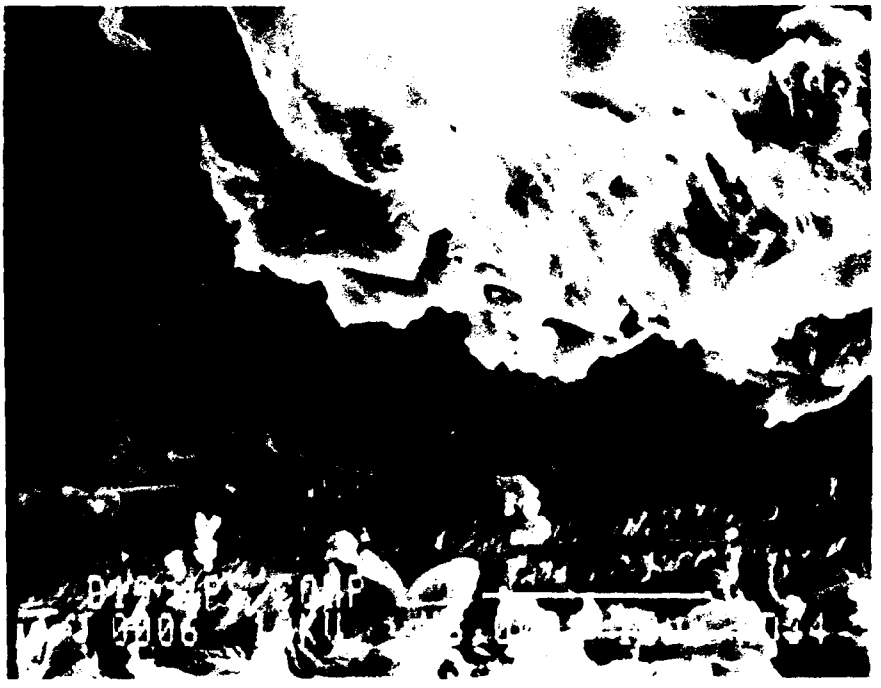


Figure 34

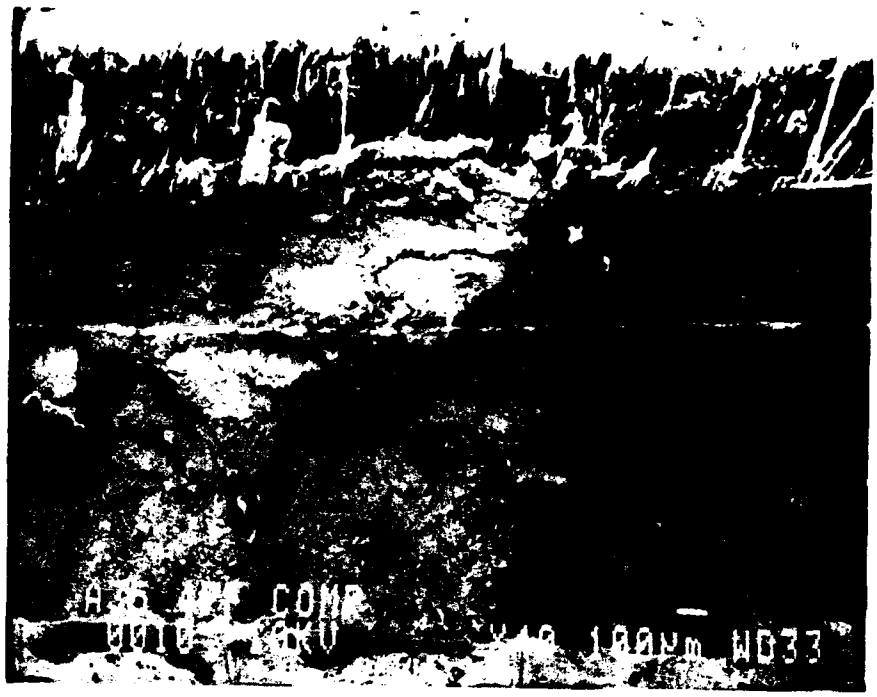


Figure 35

Figure 36

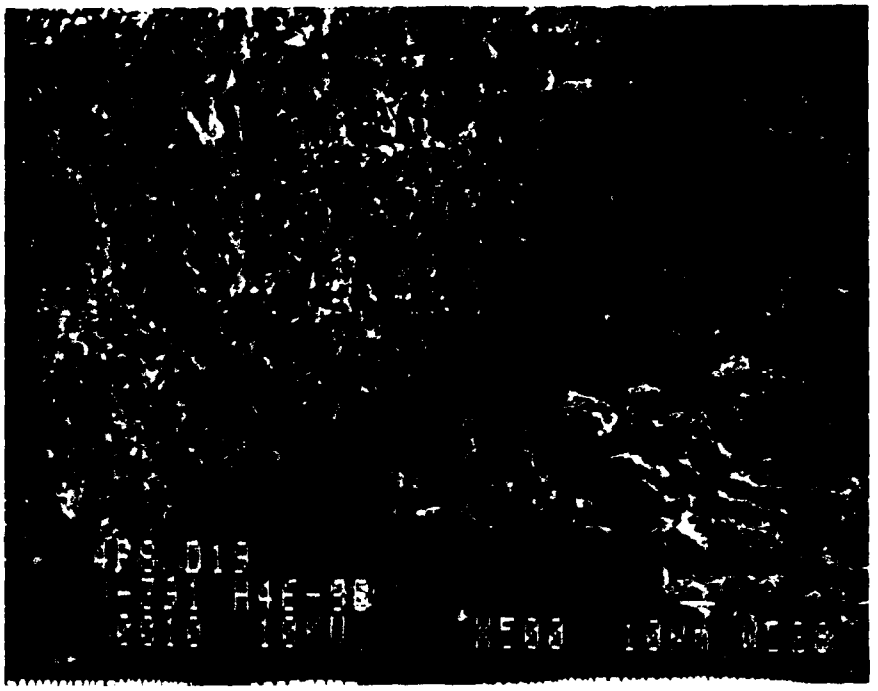


Figure 37

Comparison to Manufacturer's Data and Other BMIs

Of the manufacturer's data, only the flex test data was compatible with the test data presented here, because of differences in test methods. The manufacturer's flex strength and modulus data was slightly higher than what was measured in this work, with a room temperature dry flex strength of 1765MPa (256Ksi) from Table 8, compared to a three point flex strength of 1698MPa (246Ksi) from Table 7, but data scatter renders this difference insignificant. The hot/wet flex data was consistent with these results and strength retention was comparable. Table 9 shows mechanical properties of V-378A/T-300 from U.S. Polymeric a non-toughened BMI used at similar temperatures. The flex data compares well, with V-391 having slightly better hot/wet performance, but the interlaminar fracture toughness (G_{1C}) of V-391 at 0.27 KJ/m^2(1.57 in-lb/in^2) is more than twice that of V-378A at 0.12 KJ/m^2 (0.67 in-lb/in^2). This shows the improved toughness of V-391 over a baseline BMI, but in comparison to HTA/IM8, a thermoplastic under evaluation at MLBC[2] having a Mode I fracture toughness of 8.0 in-lb/in^2 it has only one fifth the toughness of thermoplastics. Compression after impact CAI data was used by the manufacturer to determine the fracture toughness of V-391/H46-8B. These CAI tests are rarely performed at MLBC so no data of this type was generated. If the toughness claimed by the manufacturer to be comparable to thermoplastics is actually damage tolerance as measured by CAI tests, the properties could be comparable, but data on thermoplastics to compare with the CAI values in Table 8 was not readily availiable. Comparison with F650/T-300, a toughened BMI from Hexcel, was also performed; Table 10 shows its properties as tested by MLBC in 1987 (Carlin 1987). F650 shows slightly higher strength in flex and shear tests but slightly lower fracture toughness. The largest advantage of V-391 over F650 is its two hour cure cycle with no lower temperature holds, which cuts the autoclave cure time in half, compared to other toughened BMI resin systems. Its lack of cure sensitivity is also an advantage.

[2] Data on HTA/IM8 from Capt. Karla Strong, MLBC (not yet published)

TABLE 8. Comparable Data on V-391/Hitex 46-8B from Manufacturer (Konarski 89)

UNITAPE PROPERTIES OF V-391

ON INTERMEDIATE MODULUS FIBERS

		HITEX 46-8B	IM-7
OPEN HOLE TEN. RT	MPa(Ksi)	579(84)	614(89)
OPEN HOLE T.MOD	GPa(Msi)	86(12.5)	79(11.4)
FLEX MOD.RT	GPa(Msi)	169(24.5)	158(22.9)
F MOD 121C WET	GPa(Msi)	166(24.1)	163(23.6)
F MOD 177C WET	GPa(Msi)	159(23.0)	148(21.4)
F MOD 191C WET	GPa(Msi)	132(19.2)	139(20.1)
FLEX STR RT	MPa(Ksi)	1765(256)	1544(224)
FLEX 121C WET	MPa(Ksi)	1213(176)	1262(183)
FLEX 177C WET	MPa(Ksi)	841(122)	855(124)
FLEX 191C WET	MPa(Ksi)	738(107)	724(105)
SBS RT	MPa(Ksi)	124(18.0)	121(17.6)
SBS 121C WET	MPa(Ksi)	84(12.2)	78(11.3)
SBS 177C WET	MPa(Ksi)	55(8.0)	62(9.0)
SBS 191C WET	MPa(Ksi)	37(5.4)	53(7.7)
OHC RT *	MPa(Ksi)	400(58)	434(63)
OHC 121C WET *	MPa(Ksi)	352(51)	372(54)
OHC 177C WET *	MPa(Ksi)	338(49)	359(52)
OHC 191C WET *	MPa(Ksi)	324(47)	338(49)
CAI **	MPa(Ksi)	238(34.5)	233(33.8)
CAI ***	MPa(Ksi)	317(46.0)	296(43.0)

TENSILE, FLEX AND COMPRESSIVES NORMALIZED TO 5.2 MILS/PLY
WET = 96 HOUR BOIL
LAMINATES WERE CURED FOR 2 HOURS AT 350F.
POSTCURE = 6 HOURS AT 470F.

* OPEN HOLE COMPRESSION (+/-45,90,0,0,+/-45,0,0,+/-45,0)sym.
** BOEING COMPRESSION AFTER IMPACT AT 265 J/M2 (1500 in-lbs/in)
*** NORTHROP COMPRESSION AFTER IMPACT
AT 265J/M2 (1500 in-lbs/in)

TABLE 9. Baseline mechanical properties of a similar BMI composite, V-378 (Carlin, '87)

AFWAL-TR-87-4074

Baseline Mechanical Properties V378A/T-300

TEST	CONDITIONS	PROPERTY	VALUE	VALUE
0° 4-point flex	RT, Dry	σ	271 ksi	1870 MPa
		E	19.6 msi	135 GPa
	RT, Wet	σ	233 ksi	1610 MPa
		E	16.2 msi	112 GPa
	250 F, Dry	σ	178 ksi	1230 MPa
		E	15.0 msi	103 GPa
	250 F, Wet	σ	130 ksi	898 MPA
		E	14.2 msi	97.9 GPa
	350 F, Dry	σ	143 ksi	984 MPa
		E	14.3 msi	98.6 GPa
	350 F, Wet	σ	104 ksi	719 MPa
		E	14.0 msi	96.5 GPa
0° 3-point flex	RT, Dry	σ	270 ksi	1860 MPa
		E	14.8 msi	102 GPa
	RT, Wet	σ	254 ksi	1750 MPa
		E	17.7 msi	122 GPa
	250 F, Dry	σ	220 msi	1520 MPa
		E	17.6 msi	121 GPa
	250 F, Wet	σ	193 ksi	1330 MPa
		E	17.0 msi	117 GPa
	350 F, Dry	σ	188 ksi	1300 MPa
		E	17.6 msi	121 GPa
	350 F, Wet	σ	145 ksi	997 MPa
		E	16.4 msi	113 GPa

TABLE 9. Continued

Baseline Mechanical Properties V378/T-300 — Continued

TEST	CONDITIONS	PROPERTY	VALUE	VALUE
[±30°/90°] edge delamination	RT, Dry	σ DELAM	21.3 ksi	147 MPa
		σ ULT	29.5 ksi	203 MPa
		ε ULT	0.41%	0.41%
		E	6.33 msi	47.6 GPa
0° Tension	RT, Dry	ε	1.05%	1.05%
		E	21.6 msi	149 GPa
[±45°]	RT, Dry	G_{LT}g	0.96 msi	6.62 GPa
	250 F, Dry	G_{LT}g	0.71 msi	4.90 GPa
	250 F, Wet	G_{LT}g	0.56 msi	3.86 GPa
	350 F, Wet	G_{LT}g	0.60 msi	4.14 GPa
0° 4-point shear	RT, Dry	τ	12.3 ksi	84.8 MPa
	RT, Wet	τ	11.3 ksi	77.9 MPa
	250 F, Dry	τ	9.40 ksi	64.8 MPa
	250 F, Wet	τ	6.60 ksi	45.5 MPa
	350 F, Dry	τ	7.20 ksi	49.6 MPa
	350 F, Wet	τ	4.50 ksi	31.0 MPa
90° flex	RT, Dry	σ	10.9 ksi	75.2 MPa
		E	1.50 msi	10.3 GPa
		ε	0.72%	0.72%
Short-beam shear	RT, Dry	σ	15.4 ksi	106 MPa
	RT, Wet	σ	14.3 ksi	98.6 MPa
	250 F, Dry	σ	11.0 ksi	75.8 MPa
	250 F, Wet	σ	9.10 ksi	62.7 MPa
	350 F, Dry	σ	8.30 ksi	57.2 MPa
	350 F, Wet	σ	6.70 ksi	46.2 MPa
Mode I	RT, Dry	G_{1C}	0.67 in lb/in^2	117 N m/m^2

TABLE 10. Properties of F650/T300, a similar toughened BMI (Carlin, '87)

Mechanical Testing Results F650/T-300

TEST	CONDITIONS	PROPERTY	VALUE	VALUE	FAILURE MODE
0° 4-point flex	RT, Dry	σ	236 ksi	1620 MPa	Complex
		E	17.7 msi	122 GPa	
	RT, Wet	σ	239 ksi	1650 MPa	Complex
		E	21.4 msi	148 GPa	
	250 F, Dry	σ	183 ksi	1260 MPa	Complex
		E	18.1 msi	125 GPa	
	250 F, Wet	σ	178 ksi	1230 MPa	Complex
		E	18.0 msi	124 GPa	
	350 F, Dry	σ	194 ksi	1340 MPa	Complex
		E	18.4 msi	127 GPa	
	350 F, Wet	σ	130 ksi	893 MPa	Complex
		E	15.5 msi	107 GPa	
0° 3-point flex	RT, Dry	σ	286 ksi	1970 MPa	Complex
		E	17.6 msi	121 GPa	
	RT, Wet	σ	268 ksi	1850 MPa	Complex
		E	17.9 msi	123 GPa	
	250 F, Dry	σ	248 ksi	1710 MPa	Complex
		E	17.0 msi	117 GPa	
	250 F, Wet	σ	194 ksi	1340 MPa	Complex
		E	17.0 msi	117 GPa	
	350 F, Dry	σ	221 ksi	1530 MPa	Complex
		E	17.5 msi	121 GPa	
	350 F, Wet	σ	156 ksi	1080 MPa	Compression
		E	15.9 msi	110 GPa	

TABLE 10. Continued

Mechanical Testing Results F650/T-300 —Continued

TEST	CONDITIONS	PROPERTY	VALUE	VALUE	FAILURE MODE
0° 4-point shear	RT, Dry	τ	13.2 ksi	91.0 MPa	Shear
	RT, Wet	τ	11.0 ksi	75.9 MPa	Shear
	250 F, Dry	τ	11.1 ksi	76.5 MPa	Shear
	250 F, Wet	τ	8.20 ksi	56.5 MPa	Shear
	350 F, Dry	τ	9.00 ksi	62.1 MPa	Compression
	350 F, Wet	τ	5.60 ksi	38.6 MPa	Plas Def
90° flex	RT, Dry	σ	12.3 ksi	84.8 MPa	Tension
		E	1.60 msi	11.0 GPa	
		ε	0.78%	0.78%	
	RT, Wet	σ	10.1 ksi	69.6 MPa	Tension
		E	1.60 msi	11.0 GPa	
		ε	0.65%	0.65%	
Short-beam shear	RT, Dry	σ	16.3 ksi	112 MPa	Complex
	RT, Wet	σ	15.0 ksi	103 MPa	Complex
	250 F, Dry	σ	12.9 ksi	89.0 MPa	Complex
	250 F, Wet	σ	9.90 ksi	68.3 MPa	Complex
	350 F, Dry	σ	10.9 ksi	75.2 MPa	Complex
	350 F, Wet	σ	7.10 ksi	49.0 MPa	Compression
Mode I	RT, Dry	G_{1C}	1.14 in lb/in^2	200 N m/m^2	Mode I
[±30°/90°] edge delamination	RT, Dry	σ_{DELAM}	17.6 ksi	121 MPa	Tension
		σ_{ULT}	30.6 ksi	211 MPa	
		ε_{ULT}	0.56%	0.56%	Tension
		E	6.33 msi	43.6 GPa	
0° Tension	RT, Dry	ε	1.13%	1.13%	Tension
		E	21.7 msi	149 GPa	Tension
[±45°]	RT, Dry	G_{LT}	0.95 msi	6.55 GPa	Tension
	250 F, Dry	G_{LT}	0.89 msi	6.14 GPa	Tension
	250 F, Wet	G_{LT}	0.65 msi	4.48 GPa	Tension
	350 F, Wet	G_{LT}	0.22 msi	1.52 GPa	Tension

CONCLUSION

V-391 has proven to be an improvement over current toughened BMI systems in its processability. Handling of the prepreg with its good drape and slight tack, though less than epoxies, would allow easy layup on any tooling designed for thermoset prepreg systems. Auto mated tape laying would be more difficult. The possibility of heating the material during automated layup could make this more feasible. Resin flow during cure was not a problem and seemed to be quite controllable, though only two autoclave runs were made. The short, two hour hold in the cure cycle could allow twice as much material to be cured in the same time period, lowering the cost of processing. V-391 is said to be very forgiving (Konarski 89), with less chance of ruining an expensive batch of material because of computer and human error or autoclave failure during the cure and postcure operations.

Mechanical properties, including hot/wet performance, were not a significant improvement over current toughened BMI systems, but were comparable. Interlaminar fracture toughness, by MLBC testing methods, was good, however it is only a fraction of that of thermoplastics. The manufacturer's claim that it "...combines the toughness of thermoplastics with the temperature performance of BMI's." does not agree with MLBC toughness data. Damage tolerance, as evaluated by the manufacturer's CAI tests may be comparable to thermoplastics but compatible data is not readily available.

Based on this preliminary screening, V-391/H46-8B is a viable alternative to more brittle high-temperature composites, not only due to its toughness but its processibility as well. If the cost of the material is the comparable to other systems, it would be less expensive to process and therefore is an attractive material for further investigation.

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APPENDIX A

MOISTURE, MODULUS AND STRENGTH DATA

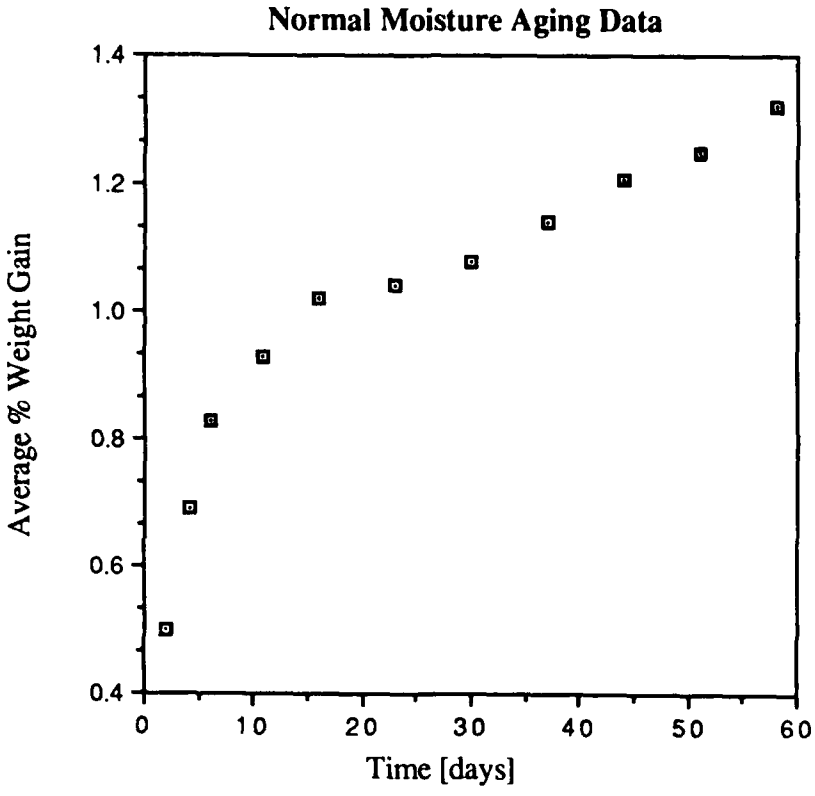

Figure 38. Plot of moisture data

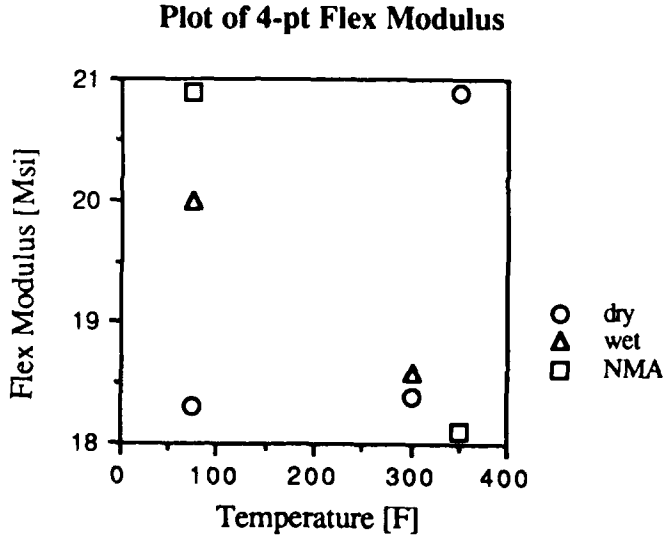

Figure 40. Plot of Modulus Data

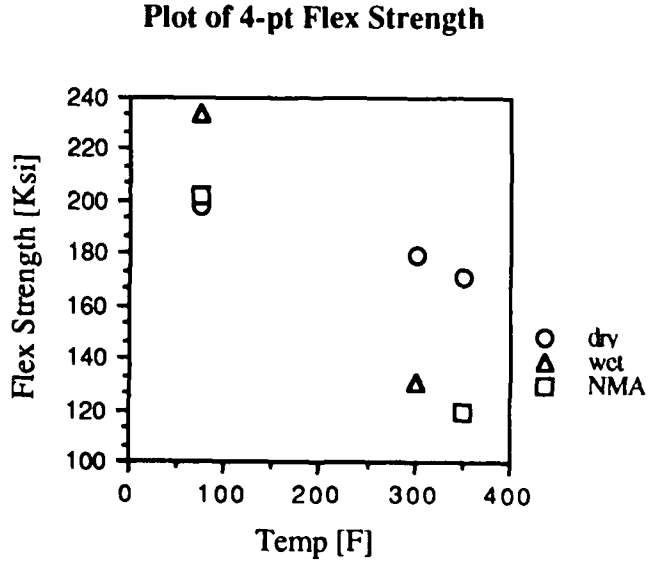

Figure 39. Plot of Strength Data

* U.S.Government Printing Office: 1990—748-056/24303